## **CLAIMS**

1. A process for preparing a compound of formula

in which R represents linear or branched  $C_1$ - $C_5$  aliphatic acyl or benzoyl, optionally substituted with  $C_1$ - $C_5$  alkyls,  $C_1$ - $C_5$  alkoxyls or halogens,

which comprises the reaction of coupling of a compound of formula

in which

R represents a linear or branched  $C_1$ - $C_5$  aliphatic acyl or benzoyl, optionally substituted with  $C_1$ - $C_5$  alkyls,  $C_1$ - $C_5$  alkoxyls or halogens,

R' represents R or a linear or branched C<sub>1</sub>-C<sub>5</sub> alkyl,

with a compound of formula

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$$(R'')_3SiO \qquad N$$

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in which R", being identical or different, represents a C<sub>1</sub>-C<sub>6</sub> alkyl or a phenyl, in the presence of a Lewis acid and in an inert organic solvent, characterized in that said Lewis acid is added at a temperature below 0°C.

- 5 2. A process according to claim 1 in which said addition of catalyst is carried out at a temperature below -10°C, preferably between approx. -15 and -20°C.
  - 3. A process according to claim 1 in which, on completion of said addition of catalyst, the reaction mixture is held further at the same temperature.

4. A process according to claim 1 in which R and R' represent acyl, preferably acetyl, and R" represents methyl.

- A process according to claim 1 in which said Lewis acid is selected from
  trimethylsilyltrifluoromethanesulphonate and tin tetrachloride, and is preferably tin tetrachloride.
  - 6. A process according to claim 1 in which said inert organic solvent is selected from chlorinated solvents or aromatic solvents, preferably chlorinated solvents.
  - 7. A process according to claim 1 in which said compound of formula II, in which R has the meanings stated above, is further submitted to a reaction of deprotection to give doxifluridine of formula I.
- 25 8. A process for the preparation of doxifluridine of formula

that comprises a process according to one of the claims from 1 to 7.